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EUROPEAN PATENT APPLICATION

21 Application number: 89810438.5

51 Int. Cl. 4: B 22 F 9/14

22 Date of filing: 09.06.89

30 Priority: 13.06.88 CH 2253/88

43 Date of publication of application:
20.12.89 Bulletin 89/51

64 Designated Contracting States: AT DE FR IT

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54 Method to simultaneously pulverize and vaporize metals into particles of varied size distribution.

57 The metal to be pulverized is subjected to an electric arc in a closed vessel containing a gas which is dissociated by the arc. The metal melts at least partially and the gas dissolves into the molten metal where it reassociates and, due to the binding energy, causes the metal to evaporate. Simultaneously, some portions of molten metal migrate by convection to cooler zones whereby oversaturation by the gas occurs which results into bubble formation. The bubbles burst and metal particles are spattered away, the rates of both evaporation and spattering depending on the temperature differences of the hotter and lesser hot zones. Pulverization is periodically interrupted to periodically disturb thermal and dynamic operative conditions which evolve to equilibrium.

EP 0 347 386 A1

Description

METHOD TO SIMULTANEOUSLY PULVERIZE AND VAPORIZE METALS INTO PARTICLES OF VARIED SIZE DISTRIBUTION

The present invention concerns a method to simultaneously pulverize and vaporize a metal or an alloy into particles of varied ranges of size distribution, this being by means of an electric arc (plasma) acting on said metal or alloy in a pulverization enclosure under an atmosphere of a gas which becomes activated, e.g. It dissociates, when subjected to the effect of said plasma of the electric arc. In a method of this kind, the metal subjected to the electric arc melts at least partly (for instance the surface of the metal becomes fluid) and the activated gas dissolves in the molten metal, especially in the hotter area thereof. After dissolving, the dissociated atoms of the activated gas bind again and the released energy causes the molten metal to boil and evaporate. When the arc power is narrow and concentrated on a relatively small portion of the sample surface, the vapor evolution is very dense and this vapor undergoes condensation to sub-micron particles (fumes or dust) which can be collected. On the other hand, when heated, the molten metal is subject to convection displacements whereby the dissolved gas moves along to somewhat cooler areas of the sample (cooler because they avoid the direct action of the arc). In these cooler areas, the vaporization is little or nought and hence the external pressure dwindles and the dissolved gas can suddenly escape by building large bubbles which burst and sputter the metal into particles larger than the dust particles which result from metal vapor condensation. The newly formed larger particles can be of the order of several μm to several hundreds of μm .

It should be noted that, depending on the arc operating conditions and other operating parameters such as electrode and sample dimension, kind of metal to be reduced to powder, and the like, one may favor evaporation (production of very fine particles of a μm or less) at the expense of sputtering or vice-versa, i.e. promote bubble formation and consecutive formation of larger particles (spattering). Hence the energy density of the arc (and consequently the localized temperature elevation) reflects on the fumes and larger particles distribution. With a short, narrow and dense arc, metal vaporization occurs mostly; with a larger and slighter arc (less stable also), larger particles resulting from spattering are mostly produced.

The aforementioned general technique has been disclosed in many publications, inter-alia the following documents : US-A-4,376,740 (NAT. RES. INST. METALS) discloses a method in which a hydrogen stream is activated by an electric arc or a plasma and discharged toward a molten metal or alloy mass which results into pulverization of this metal to particles of sizes below 10 μm . As activated hydrogen sources, elemental hydrogen, NH_3 , CH_4 , C_2H_6 , propane and ethylene are mentioned. The activated hydrogen can be diluted in a noble gas such as He, Ne, Ar, etc. The metal powder is wiped off by the hydrogen stream to a duct connected to a sedimentation enclosure in which it collects.

Document US-A-4,482,134 (NAT. RES. INST. METALS) discloses a method and a device similar to that of the previous document but comprising, in addition, appropriate means for preventing the newly formed particles from agglomerating and forming aggregates exceeding 5 μm . This consists in rapidly cooling the newly formed particles by means of an air stream and to drive them by this stream into a container where they settle by sedimentation.

Document US-A-4,689,075 concerns a method for making ultra-fine powders from metals or ceramics, i.e. particles of size definitely below 1 μm . In this method, at least two different materials are pulverized simultaneously using electric arcs operating under gas pressures ranging from 50 Torr to 3 atmospheres, the selected gases being hydrogen, nitrogen, oxygen or mixtures thereof. The newly formed particles from both or more materials are taken up in a gas stream whose turbulent motion ensures proper mixing of the different particles as they are still suspended in the gas. From the operating conditions disclosed in this document, it would appear that the particles generated result from an evaporation process rather than from the spattering of bubbles in the molten material.

Similarly, document JA-59-190.302 (DAIDO TOKUSHUKO) describes the manufacture of superfine particles by subjecting a metal, a semi-conductor material or an alloy to melting under the electric arc in the presence of hydrogen, or nitrogen, or a hydrogen generating gas. From this document, the molten metal is held in a cylinder arranged coaxially with an arc generating electrode and provided with a piston which operates on the molten metal for keeping it at constant level with respect to the electrode; hence, the voltage, the length and the current of the electric arc are under constant control.

Although the techniques known so far are interesting, they have the following drawbacks : despite the presence of means for cooling the holder substrate that supports the metal to be pulverized, the violent heat effect to which the latter is subjected will generate, after a relatively short working period of less than a minute to several minutes, a set of thermal and chemical equilibrium conditions which inhibit pulverization, hence the latter slows down or even stops completely. Asking about the detailed reasons for this inhibition, one can theorize on the temperature differences between the gas dissolving areas and the gas escaping areas; this difference progressively reduces as the reaction proceeds which possibly decreases the gaseous exchanges between the areas.

Hence, the wider the area of the molten metal (which is normally so with an uninterrupted application of the arc), the rarer becomes the concentration of the dissolved gas in the cooler region surrounding the arc; consequently, bubble formation will slow down with time as well as spattering.

On the other hand, the metal vapor which emanates from the metal area directly under the arc will

progressively bring about a screening effect thereto and prevent the activated gas from being in contact with the molten metal. Hence the rate of evaporation also decreases progressively.

Moreover, in view of the foregoing progressively occurring modification, the pulverization operating conditions will vary and, as a consequence, the distribution of the production of the very fine particles (which result from fumes condensation) and that of larger particles (spattering) is progressively shifted from where it was at the beginning. For instance, in the earlier moment of operation, one may obtain nearly exclusively a very fine dust resulting from evaporation while, later on, a mixture of fine dust and spattered larger particles. Actually, as far as one knows, the methods of the prior art did not allow by only setting up appropriately the operating conditions to selectively favour the production of either very fine dust issued from evaporation, or spattered particles, or both. The present inventors have attempted to remedy the aforementioned drawbacks and, as a consequence, they invented the method summarized in claim 1.

In this connection, the teaching of document GB-A-2,176,582 (DAIDO TOKUSHUKO) should be mentioned. This document discloses an apparatus for pulverizing molten metal by the periodic application of a plasma blowing torch. This torch consists of a ring-like nozzle which ejects an activatable gas stream slantwise toward a ring of metal contained in a toroidal pot located coaxially below the nozzle. Hence the gas stream is in the form of a frusto-conical curtain which continuously, strikes the metal in the pot. An electric arc is generated locally in the gas between a ring electrode in the nozzle and the metal to be pulverized by a magnetic field which acts on a portion of the gas curtain and which can be progressively rotated along the ring by conventional means. The metal in the pot heats up and melts locally so that part of the arc activated gas dissolves therein. As the arc moves away, the portions of molten metal no longer under the arc cool slightly, become oversaturated with dissolved gas and erupt with production of spattered particles which are wiped off by the annular gas stream. Hence, the method disclosed in this document does not involve simultaneous controlled evaporation of the molten metal like in the present invention. Furthermore, in the apparatus of the reference, the stream of gas is blown permanently on the perimeter of the metal to be pulverized which, operationally, is not economical. In contrast, in the present invention, no gas stream is projected over the molten metal, but an alternation of "live" or active periods with "dormant" or quiescent ones is brought about, i.e. periods in which the metal is subjected to the electric arc and will progressively tend to dynamic thermal and chemical equilibrium successively alternate with "passive" periods in which the metallic dust of evaporation is eliminated and during which the metal cools off sufficiently for restoring the initial chemical and thermal conditions which promote effective pulverization at the beginning of the next period. Moreover, steps can also be taken to remove the metallic dust or fumes resulting from condensation of the vapors from the operating zone under the electric arc, this being possible, for instance by condensing the dust on a surface with particle attracting properties or by carrying away the particles and allowing them to settle into an appropriate receptacle. Hence, although the initial yield of the pulverization operation is progressively perturbed and inhibited toward the end of a previous live period by the presence of metal vapor in the zone directly influenced by the electric arc, favorable operating conditions are renewed during the quiescent period and fully restored at the beginning of the next live period.

The duration of the live and dormant periods is quite variable and depends on many features, namely the kind of metal to be pulverized, the electric arc operating parameters, the size of the metal particles and the volume or weight distribution ratio between the particles of micronic or overmicronic size (produced by spattering) and the sub-micronic particles which result from metal vapor condensation.

To properly select the duration of the live and dormant periods, care should also be taken of the kind of particles desired. Thus, if only sub-micronic particles (fumes) are desired, the live periods will end before spattering occurs, i.e. before the phenomena involving dissolved gas evolution become significant.

If spattering must be favored at the expense of fumes production, the arc parameters will be adjusted correspondingly, i.e. energy density, arc length, arc intensity and so on, so that the evaporation phenomena are minimized as much as possible and the duration of the live phase is determined in function to the evolution of the operation, e.g. the course of the pulverization progressive inhibition.

In general, the live and dormant periods are in the order of a few seconds to a few minutes, e.g. 5 sec. to 5 min. and are set by experiment depending on the needs. Naturally the duration of the live and dormant phases can be identical or different.

In order to temporarily remove the sample of metal to be pulverized from the effect of the electric arc, one may either cut off the power to the electric arc, or one may physically withdraw the sample to put it out of reach of the arc. Both embodiments are disclosed hereafter with reference to the annexed drawing.

Fig. 1 is a schematic representation of an apparatus for pulverizing a metal by the action of an electric arc in an atmosphere of hydrogen diluted in an inert gas.

Fig. 2 is a highly enlarged schematic illustration of a detail of the pulverization process.

Fig. 3 is an enlarged schematic representation of a modification of the supporting structure for the sample to be pulverized.

The apparatus pictured in fig. 1 comprises a gas tight pulverization enclosure 1 housing a supporting structure 3 of a metal sample 3 to be pulverized. In facing registration with this sample, there is a cathode 4 of zirconia/tungsten or thoriated tungsten (or any electrically conducting refractory material). This cathode 4 is provided with a liquid cooling circuit 5, with a power line 6 connected to a power supply not represented and with an element 7 connected to a lever 8 rocked at position 9 for raising and lowering the cathode 4.

The enclosure 1 further comprises a receptacle 11 which surrounds holder 3, itself cooled by a liquid circuit

10, which receptacle 11 serves to collect the particles 12 of the pulverized metal that settle to the bottom thereof. The receptacle is also cooled by a liquid circuit 10a, and the enclosure is also cooled by circuit 10b.

The enclosure 1 further comprises a gas input 12 and an output 13 connected to a decantation element not represented here (for instance a cyclone) to retain by deposition the ultra-fine dust resulting from direct evaporation. The flow rate of this gas is very low (1-20 l/hrs approximately) and exerts no mechanical or cooling action on the metal being pulverized. This gas can be hydrogen or a mixture of hydrogen and a noble gas such as argon, e.g. a 5-50 % (v/v) H_2/Ar mixture. The electrode 4 (cathode) is connected to a negative terminal of an electric generator while the enclosure is connected to the positive terminal.

For starting the present apparatus, the cathode is lowered toward the sample 3 by means of the lever elements 7, 8 so as to trigger the development of an arc 14 between the electrode 4 and the sample 3 (see fig. 2). The metal heats up, it melts and starts evaporating in the space directly subjected to the arc as indicated by arrows 15. The gas which fills the enclosure under reduced pressure (e.g. 100-800 Torr) gets activated by dissociation and starts dissolving into the molten metal, more particularly in the space 14 which belongs to where the arc strikes the metal; then the dissolved gas is driven by the thermal displacement of the molten metal outside the hotter region 14 and there it starts forming bubbles 16 because of oversaturation. The bubbles near the surface of the molten metal burst and spatter in the form of droplets or particles (often of spherical shape) the size of which is of the order of a few fractions of a μm to several tens or hundreds of μm (or more). These particles solidify upon cooling and accumulate (12) in receptacle 11. Fumes 15 which form upon condensation in the enclosure of the metal vapor evolved in the space directly subjected to the arc stroke are taken to outlet 13 and are collected as indicated above in sub-micronic dust form. As a variant, a screen can be interposed on the path of the fumes, for instance a removable wall capable of collecting the dust by electrostatic means wherefrom it can be gathered later. The present apparatus is therefore capable of selecting metal powders depending on particle size. It should be noted that if, when the arc is disconnected, the tip of the cathode is dipped into the molten metal, a metal film adheres to the cathode. Then, when the arc is switched on again, one may transiently invert the direction of current (inverted polarity) which brings about instant pulverization of the metal film adhering to the cathode.

Instead of periodically cutting on and off the arc, one may temporarily withdraw the sample from the action of the arc by taking it away physically.

Fig. 3 represents a holder 22 of conductive refractory material comprising a rod 23 which can be rotated by some common mechanism (not shown here). This holder comprises two recesses 24a, 24b each of which contains a sample of metal to be pulverized. Each recess can be put in turn in facing relation with the electric arc 14 (exactly like the one sample 3 of Fig. 1), so that when one of the sample is the live phase, the second is quiescent. Transfer from one position to the other can be progressive (slow continuous rotation) or with substantially no or little transition (fast 180° step).

Of course, the number of recesses carrying metal samples can exceed 2 (it can be 4, 6 or more), or the sample can be a full ring (as in GB-A- 2.176.502) which rotates regularly, each portion of which enters repeatedly the active area of the arc and from there moves away for a next turn.

Many metals can be powdered in the present method including common and precious metals such as Al, Fe, Ni, Cu, Mn, Cr, W, Ni, Ag, Au, Pd, Pt, Rh, etc. and the alloys of two or more of these metals.

It should be noted that, depending on the operating conditions, spattering can be favoured at the expense of evaporation, this being so for instance with repeated alternate melting and solidification of the external metal layer which promotes over-saturation by the dissolved gas; it is therefore advantageous when this kind of regime is desired to have rather short live and dead periods, i.e. in the order of 10 to 40 sec. Also in this case the energy density of the arc should be adjusted correspondingly.

The actual pressure in the enclosure also has an influence on the respective contribution of evaporation and spattering; lower pressures favor evaporation; high pressures, e.g. 730 - 750 Torr favor spattering.

The following examples illustrate the invention.

Example 1

A device was used in conformity with the apparatus illustrated schematically by fig. 1.

The enclosure 1 is a Balzers sealed enclosure of $1 m^3$, water-cooled.

The crucible, cooled by water, is of copper and has a diameter of 6,3 cm. It is connected to the enclosure by a vertical pillar. The electrode is of thoriated-tungsten; it is water-cooled and its diameter is 6,4 mm. The enclosure is evacuated by two pumps Balzers, a primary pump and a diffusion pump. The gas is a 20 to 50 % (v/v) mixture of H_2 and argon. The generator can provide an arc of about 50 to 200 A.

For the experiments, a sample of metal flakes (about 5 to 30 g) was placed in the crucible; then, after flushing the enclosure with argon, it was filled with the above gas mixture under 740 Torr; the flow rate was very slow (about 5 - 10 l/hr removed by output 13 and replaced correspondingly by input 12). The gases are provided from usual pressure cylinders equipped with pressure reducers and rotameters. After starting the arc by bringing the electrode near the crucible (5 mm), the electrode was withdrawn slightly (gap 10 mm) and the current was adjusted to a value below the priming value. The arc remained on for the duration of a live period. Then the current was cut off and the whole was left to cool for the duration of the quiescent period; after that, the arc was started again and so on. The very fine powder (below $1 \mu m$) was collected in a cyclone apparatus adapted to the exhaust 13, whereas the coarser powder (particles between about 1 and $100 \mu m$) deposited into vat 11.

Results (order : control, then test) concerning pulverization of silver, palladium and an Ag/Pd (70/20) alloy are reported in the Table hereafter.

It should be noted that the total duration of the operation is given in the Table in terms of the number of working cycles, i.e. the number of PA's (PA = active periods) in minutes. The controls experiments were effected using an uninterrupted active period the duration of which corresponds to the total of the PA's of the tests. The figures between brackets in the two last columns express approximately the gain (in terms of pulverization rate) which results from operating according to the invention.

TABLE

Metal	I(A)	%H ₂	Nb. of cycles	Time (min)		Rate : mg/sec improvement coefficient	
				PA	PP	fine particles (fumes)	large particles (spatter)
Pd	160	20	-	-	-	2.7 -	6.3 -
	120	20	4	0.5	0.17	14.0 (4x)	14.0 (2x)
	160	40	-	-	-	2.7 -	2.2 -
Ag	120	40	4	0.4	0.17	21.0 (8x)	14.0 (6x)
	150	20	-	-	-	1.7 -	8.7 -
	120	20	12	0.5	3.5	9.9 (6x)	11.2 (1.3x)
	120	40	-	-	-	11.5 -	21.4 -
	120	40	8	0.5	3.5	13.5 (1x)	13.2 (0.7x)
Ag/Pd	180	20	-	-	-	1.1 -	≈ 1.0
	150	20	5	0.5	0.17	7.4 (7x)	3.7 (4x)
	150	40	-	-	-	4.3 -	1.2 -
	120	40	5	0.5	0.17	11.7 (3x)	2.6 (2x)

PA = Active period

PP = Passive period

Example 2

A 50/50 by weight Iron/nickel alloy was powderized under the following conditions.

Gaseous mixture H₂/Ar 25/75 (v/v); 740 Torr.

Tungsten electrode (2% of thorium), diameter 6.3 mm, placed at 5 mm from the sample (active period).

Crucible of annealed copper, water-cooled.

Arc of 30 V, 150 A- Active and passive periods, 1 min. each.

Under these conditions, the formation of very fine particles (of the order of 0.5 to 1 μm) takes place with a rate yield better than 200 g/hr. As comparison, by operating continuously (without quiescent periods), the yield was only 94 g/hr.

Example 3

There was proceeded as indicated above, using a 50/50 by weight Cu/Ni alloy. The operating conditions were as follows

Gaseous mixture H₂/Ar 30/70 (v/v); 750 Torr.

Electrode as in Example 3, set at 3 mm from the sample metal contained in a copper crucible cooled with water circulation.

Arc 25 V, 180 A. Active and dead periods as in Example 3.

Results : formation of mostly very fine particles (0.15 - 1.7 μm) at rate of 200 g/hr. A control test (no dead periods) gave only 96 g/hr.

Example 4

According to the present invention, the gas used in the enclosure can also be of a kind that may react with the molten metal to be powderized, i.e. the operation provides a powder of a compound of the metal and the gas. This is so when titanium is pulverized under a 90/10 (v/v) mixture of nitrogen and hydrogen under 740 Torr, 40 V, 200 A, the electrode being as in the foregoing cases but with a diameter of 3.5 mm. Active and dormant periods, 1 min. each. Under these conditions, the rate yield of titanium nitride (particles of mostly about 0.1 μm) was 20 g/hr. The control experiment effected continuously gave only 5 g/hr. of the same material.

Example 5

Copper was converted to powder under the following conditions :

H₂/Ar 30/70 (v/v), 740 Torr; 20 V, 150 A; tungsten electrode with 1% of Th, diameter 6.4 mm placed at a distance of 8 mm from the sample; graphite crucible; active periods 1 min; dead periods 1.5 min. There were thus obtained 46 g/hr. of copper particles of size mostly between 5 and 50 μ m. In comparative continuous operation, the yield was below 2 g/hr.

Using the same metal but setting the conditions as given below H₂/Ar 10/90 (v/v), 750 Torr; 20 V, 180 A; tungsten electrode with 2% of Th, 3.5 mm, placed at 3 mm of the metal sample; graphite crucible, gave particles of the order of 0.1 μ m and less with a yield of 30 g/hr. A comparative experiment effected continuously gave 5 g/hr.

Example 6

An Al/Fe, 94/6 by weight, was powderized into particles with average size above 2 μ m, the production rate exceeding 50 g/hr, by using the following conditions :

H₂/Ar 20/20 (v/v), 740 Torr; 17 in, 120 A; tungsten electrode with 2% of Th, 6.3 mm of diameter, placed at 8 mm of the substrate; copper crucible. Live and dead periods, 1 min. each. By operating under a continuous regime, the production of powder was insignificant.

The foregoing examples demonstrate that, in addition to the key factor of discontinuous operation by which the pulverization yield can be dramatically enhanced, the average size of the powder particles can be varied by modifying the various parameters that control pulverization, e.g. the gas pressure in the enclosure, the material of the electrode and its size as well as the distance between the electrode and the metal sample, and also the intensity of the electric arc. The larger the surface of the sample under the effect of the arc, the greater the proportion of dust which results from vaporization. At the limit, if the metal melts easily and the live period is long with a very strong arc, all the available surface of the metal becomes melted and the production of fumes (very fine particles) predominates. Furthermore, as explained herebefore, one may control as desired the operative parameters for obtaining either mostly fumes (fine particles) or mostly spattered particles, or both.

Claims

1. A method to powderize under an electric arc a metal sample supported by a holder in a closed vessel containing a gas which undergoes activation by dissociation when subjected to the effect of the electric arc, in which the metal melts at least partly under the effect of the arc method, the dissociated gas dissolves into the molten metal preferably in the hotter area thereof then it reassociates and, due to the release of binding energy, the molten metal starts to evaporate and to evolve metal fumes while a portion thereof moves from the hotter area to a somewhat lesser hot zone where the arc pressure is less and in which the solubility of the gas becomes insufficient, whereby the gas escapes from the oversaturated metal by forming bubbles which burst and spatter metal particles, the rate of this process depending on the temperature difference of the hotter and cooler zones, the method being characterized by carrying out cyclically the steps of :

(i) applying the arc over the metal for a "live" period in which the reaction evolves to a dynamic equilibrium, i.e., the release of the metal vapor decreases the concentration of the activated gas in contact with the molten metal and the standing operating conditions become insufficient to sustain further efficient spattering and vaporization; then

(ii) removing the metal from the influence of the arc for a period called "quiescent" or "dormant" in which the metal vapor is collected, the sample cools and a thermal unbalance between the hotter and cooler zones of the system reforms, then the initial operational conditions of step (i) are resumed.

2. The method of claim 1, in which the metal vapor that escapes from the hotter area condenses to extremely fine dust particles which spread in the atmosphere of the vessel, characterized in eliminating this dust by causing it to deposit or agglomerate so that it can be collected.

3. The method of claim 1, in which the duration of the live and quiescent periods is equal or different and range from 5 seconds to 5 minutes.

4. The method of claim 1, in which the sample is removed from the influence of the arc by switching off the arc power supply or by physically withdrawing the sample from active reach of the arc.

5. The method of claim 4, in which the sample to be pulverized is placed at the periphery of a horizontal turntable and, to remove the sample from the arc at the end of said live period, the turn-table is rotated by a given angular value.

6. The method of claim 4, in which the turn-table comprises two or more samples distributed evenly, and in which each sample comes, in turn, in facing relation with the arc for a live period, the other samples being then in a quiescent period.

7. The method of claim 4, in which the sample is at the periphery of a supporting turn-table so as to form a crown or ring, and in which this crown is rotated so that each element of the metal to be pulverized becomes progressively subjected to the effect of the electric arc and, then, removed therefrom.

8. The method of claim 4, in which when the power of the arc is switched off the tip of the cathode is

dipped into the molten metal of the sample, whereby a film of solidified metal will deposit to this tip, then when the arc is switched on at the start of the next live period the current is temporarily reversed which causes the metal of said film to be efficiently pulverized.

9. The method of claim 1, in which the gas in the vessel is diatomic, this gas being used alone or in admixture with an inert gas.

10. The method of claim 9, in which the gas is hydrogen in admixture with argon, the proportion (v/v) being 5% to 50%.

11. The method of claim 10, in which the pressure of the gas in the vessel is 700 to 760 Torr.

12. The method of claim 1, in which the spattered particles size range from about a fraction of a μm to several hundreds of μm .

13. The method of claim 2, in which the size of the particles of the dust which results from condensation of the metal vapors is in the sub-micronic range.

14. A device for carrying out the powder making method of claims 1 - 13, characterized in comprising a sealed pulverization enclosure provided with an electroconductive holder for supporting the metal sample to be pulverized, an electrode in registration with this metal sample holder, this electrode being made of refractory electroconductive material, movable means for withdrawing the electrode from the sample to be pulverized (or vice-versa) and to bring it again near to it afterwards, means to introduce gases into the enclosure and to sweep it therewith, means to collect the metal powders resulting from vaporization and pulverization, and means to generate an electric arc between the electrode and the sample on the holder.

15. The device of claim 14, in which the means for collecting the metal powder also enable to effect segregation depending on particle size.

16. The device of claim 15, in which the means for collecting larger particles consist of a vat coaxial with the sample holder in which the particles accumulate by gravity.

17. The device of claim 15, in which the finer particles are removed by the gas which circulates in the enclosure, the latter comprising in the path of the gas, either a screen on which the particles will deposit, or a cyclone in which the particles will collect by sedimentation.

FIG. 1

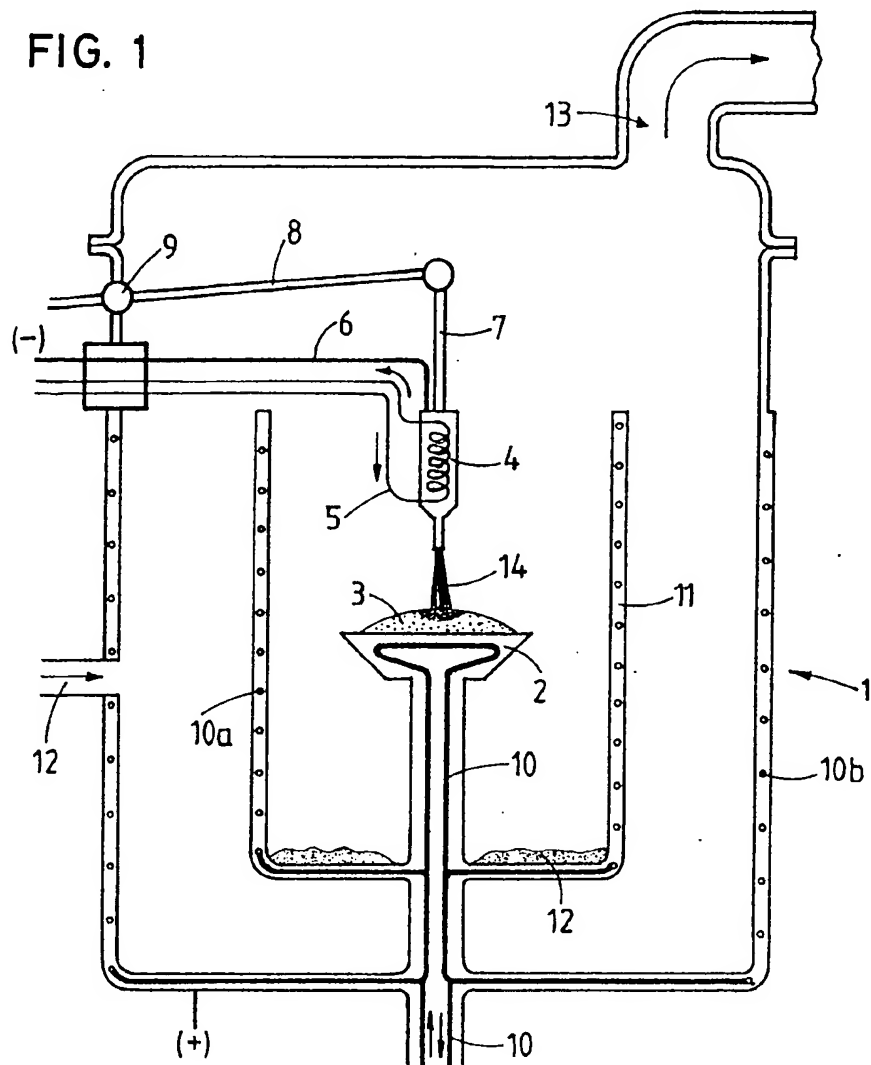


FIG. 2

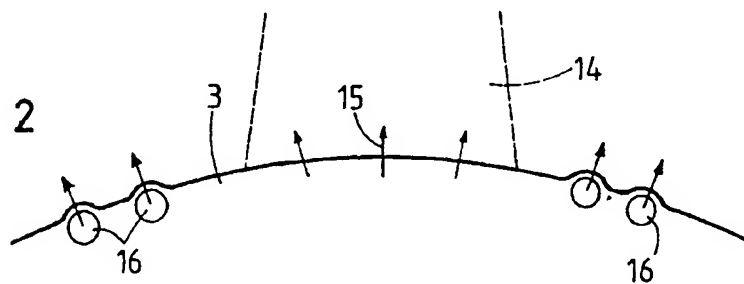
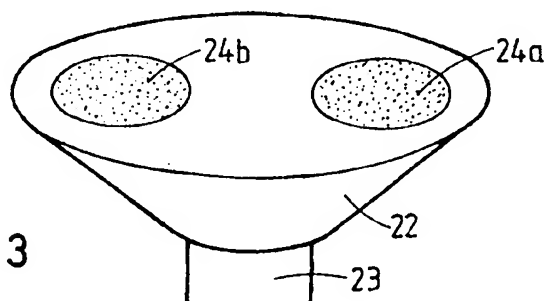


FIG. 3





European Patent
Office

EUROPEAN SEARCH REPORT

Application Number

EP 89 81 0438

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. 4)
X	GB-A-2 176 582 (DAIDOTOKUSHUKO K.K.) * Page 2, line 29 - page 3, line 118 *	1-17	B 22 F 9/14
A	WO-A-8 404 065 (NUCLEAR METALS INC.) * Claim 1; page 14, lines 1-8 *	5-7	
A,D	US-A-4 689 075 (M. UDA et al.) * Column 4, example 1; claim 1 *	9-13	
A	FR-A- 550 891 (G. BOIVIN) * Page 1, lines 21-28,43-47; abstract *	3-4	
A	FR-A- 937 763 (LASARENKO et al.) * Abstract 2b *	8	
A	US-A-4 238 427 (D.C. CHISHOLM) * Claims 1,8 *	1-2	
The present search report has been drawn up for all claims			TECHNICAL FIELDS SEARCHED (Int. Cl. 4)
			B 22 F 9/14
Place of search THE HAGUE		Date of completion of the search 28-08-1989	Examiner SCHRUERS H.J.
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons * : member of the same patent family, corresponding document			

EPO FORM 1503 01/82 (P0401)